

95-252433/33

E23 G08

JNS 93.12.08

*JP 07157681-A

E7-D12, 25-E1) G(6-G5)

KONICA CORP

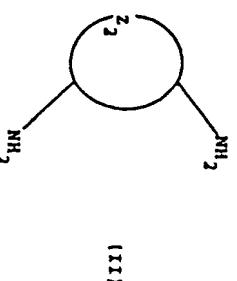
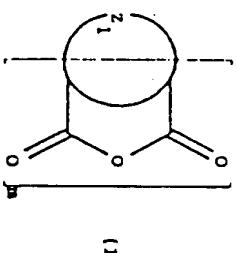
93.12.08 93JP-308212 (95.06.20) C09B 67/20, C07D 471/04, 487/04,

487/06, 487/22, C09B 57/00 // C03G 5/06

Prepn. of organic pigments used in photosensitive material for electrophotography - comprises preparing condensed imidazole pigment by dehydration-condensation of aromatic carboxylic acid anhydride and aromatic diamino cpd. using pyrimidine deriv. as solvent

C95-115387

Prepn. of organic pigments (A) comprises using a cpd. of formula (IV) as a reactive solvent in the prepn. of a condensed imidazole pigment of formula (III) by the dehydration-condensation of an aromatic carboxylic acid anhydride of formula (I) and an aromatic diamino cpd. of formula (II):



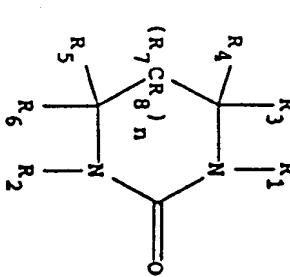
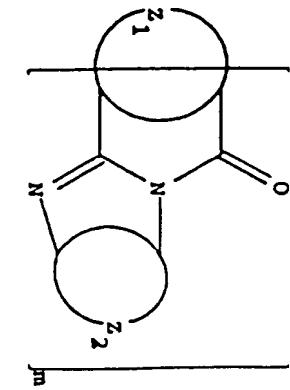
JP 07157681-A +

ADVANTAGE

The prepn. provides the pigment at a low temp. (e.g. 80-180°C). The material has high sensitivity, providing more rapid outputs without adverse environmental affect.

PREFERRED MATERIAL

Z_1 in formula (I) is a **perylene ring** or (N-alkyl) **perylene-3,4-dicarboxyimide ring**. (MO) (2) pp063DwgNo.0,0)



Z_1, Z_2 = substid. or unsubsid. aromatic ring;
 m = 1 or 2;
 R_1-R_6 = H, alkyl or aryl; and
 n = 0 or 1.

Also claimed is a photosensitive material for electrophotography contg. the organic pigment.

USE

The photosensitive material is used in printers, copy machines,

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(C) WPI / DERWENT

AN - 1995-252433 [33]

AP - JP19930308232 19931208

CPY - KONS

DC - E23 G08 P84 S06 T04 W02

FS - CPI;GMPI;EPI

IC - C07D471/04 ; C07D487/04 ; C07D487/06 ; C07D487/22 ; C09B57/00 ;
C09B67/20 ; G03G5/06

MC - E07-D12 E25-E01 G06-G05

- S06-A01A1 T04-G04C W02-J02B2

M3 - [01] F011 F012 F013 F014 F015 F016 F017 F019 F523 F543 G010 G019 G020
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M510 M521 M530 M531 M532 M533 M540 M781 M903 M904 Q346 R043;
9533-D4201-UM4 - [02] D011 D019 D021 D022 D023 D029 E350 G010 G019 G100 J5 J522 L9 L941
L999 M112 M210 M211 M240 M280 M281 M282 M283 M320 M412 M511 M520 M530
M532 M540 M720 M903 M904 N205 N209 N241 N261 N306 N331 N332 N513 Q346
R043 W001 W003 W030 W334 W543; 07430 09713 66475; 9533-D4202-P- [03] D011 D012 D019 D021 D022 D023 D029 E330 E350 G010 G019 G100 H181
H201 J5 J521 J522 L9 L941 L999 M112 M210 M211 M213 M231 M240 M273 M280
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N209 N241 N261 N306 N331 N332 N513 Q346 R043 W001 W003 W030 W334 W543;
04174 07429 07564 07663 14050 66509 73355 73724 73731; 9533-D4203-P

PA - (KONS) KONICA CORP

PN - JP7157681 A 19950620 DW199533 C09B67/20 021pp

PR - JP19930308232 19931208

XA - C1995-115387

XIC - C07D-471/04 ; C07D-487/04 ; C07D-487/06 ; C07D-487/22 ; C09B-057/00 ;
C09B-067/20 ; G03G-005/06

XP - N1995-195116

AB - J07157681 Prepn. of organic pigments (A) comprises using a cpd. of
formula (IV) as a reactive solvent in the prepn. of a condensed
imidazole pigment of formula (III) by the dehydration-condensation of
an aromatic carboxylic acid anhydride of formula (I) and an aromatic
diamino cpd. of formula (II): Z1, Z2 = substd. or unsubstd. aromatic
ring; m = 1 or 2; R1-R6 = H, alkyl or aryl; and n = 0 or 1.

- Also claimed is a photosensitive material for electrophotography
contg. the organic pigment.
- USE - The photosensitive material is used in printers, copy machines,
facsimiles, etc. using an electrophotographic system.
- ADVANTAGE - The prepn. provides the pigment at a low temp. (e.g.
80-180deg.C). The material has high sensitivity, providing more rapid
outputs without adverse environmental affect.
- (Dwg.0/0)

CN - 9533-D4201-U 9533-D4202-P 9533-D4203-P

IW - PREPARATION ORGANIC PIGMENT PHOTOSENSITISER MATERIAL

ELECTROPHOTOGRAPHIC COMPRIZE PREPARATION CONDENSATION IMIDAZOLE
PIGMENT DEHYDRATE CONDENSATION AROMATIC CARBOXYLIC ACID ANHYDRIDE
AROMATIC DI AMINO COMPOUND PYRIMIDINE DERIVATIVE SOLVENT

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NC - 001

OPD - 1993-12-08

ORD - 1995-06-20

PAW - (KONS) KONICA CORP

RRL - 07430 09713 66475 04174 07429 07564 07663 14050 66509 73355 73724 73731

TI - Prepn. of organic pigments used in photosensitive material for
electrophotography - comprises preparing condensed imidazole pigment
by dehydration-condensation of aromatic carboxylic acid anhydride and
aromatic diamino cpd. using pyrimidine deriv. as solvent

PATENT ABSTRACTS OF JAPAN

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5

(51)Int.Cl.

C09B 67/20
 C07D471/04
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 C09B 57/00
 // G03G 5/06

(21)Application number : 05-308232

(71)Applicant : KONICA CORP

(22)Date of filing : 08.12.1993

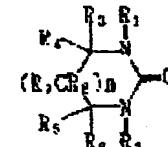
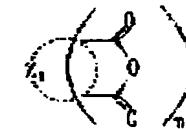
(72)Inventor : SAKIMURA TOMOO
 KINOSHITA AKIRA
 SUZUKI TOMOKO

(54) PRODUCTION OF ORGANIC PIGMENT AND ELECTROPHOTOGRAPHIC PHOTORECEPTOR CONTAINING ORGANIC PIGMENT

(57)Abstract:

PURPOSE: To obtain a high-performance condensed imidazole pigment by a low-temperature reaction by condensing a specified aromatic carboxylic acid anhydride through dehydration with an aromatic diamino compound in a specified reaction solvent.

CONSTITUTION: An aromatic carboxylic acid anhydride of formula I is condensed through dehydration with an aromatic diamino compound of formula II in a reaction solvent comprising a compound of formula III to produce a condensed imidazole pigment of formula IV. In those formulas, Z₁ and Z₂ are each a (substituted) aromatic ring; (m) is 1 or 2; R₁ to R₈ are each H, an alkyl or an aryl; and (n) is 0 or 1. Because the conventional process for producing a condensed imidazole pigment necessitates the use of a reaction at high temperatures, the produce is apt to be contaminated with impurities produced by side reactions and decomposition reactions, and expensive equipment is necessary. According to the process, a high-performance condensed imidazole pigment not contaminated with impurities can be produced by a low-temperature reaction. A highly sensitive electrophotographic photoreceptor can be provided by using this pigment.



LEGAL STATUS

[Date of request for examination]

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CLAIMS

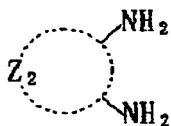
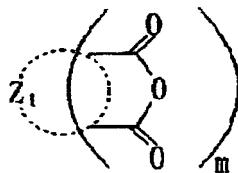
[Claim(s)]

[Claim 1] The manufacture method of the organic pigment characterized by using the compound expressed with a general formula [4] as a reaction solvent by the dehydration condensation reaction of an aromatic diamino compound expressed with the aromatic carboxylic-acid anhydride expressed with a general formula [1], and a general formula [2] in case the condensation imidazole pigment of a general formula [3] is manufactured.

[Formula 1]

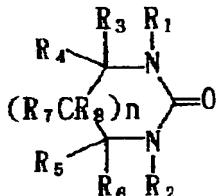
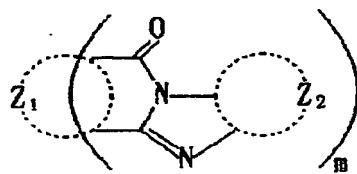
(1)

(2)



(3)

(4)



As for the inside Z1 and Z2 of a formula, the aromatic ring which is not replaced [substitution and] is expressed and m expresses 1 or 2. Moreover, R1-R8 express a hydrogen atom, an alkyl, and an aryl group, and n expresses 0 or 1.

[Claim 2] Z1 in a general formula [1] is a perylene ring or (N-alkyl) a perylene. - 3, 4, the manufacture method of the organic pigment according to claim 1 which is - dicarboxyimide ring.

[Claim 3] The electrophotography photo conductor which comes to contain the organic pigment characterized by using the compound expressed with a general formula [4] as a reaction solvent by the dehydration condensation reaction of an aromatic diamino compound expressed with the aromatic carboxylic-acid anhydride expressed with a general formula [1], and a general formula [2] in case the condensation imidazole pigment of a general formula [3] is manufactured.

[Formula 2]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] this invention relates to the manufacture method of a condensation imidazole pigment, and application to an electrophotography photo conductor.

[0002]

[Description of the Prior Art] The spread of the copying machine which outputs using an electrophotography method, a printer, facsimile, etc. has a remarkable thing, it can output more quickly and more beautifully, and what has the smaller impact to environment is called for today.

[0003] Although the material of inorganic systems, such as SeAs and CdS, has been conventionally used for the electrophotography photo conductor, the material of the organic system which has bigger possibility in points, such as the versatility of material selection, a low cost, low-pollution nature, and an ease of handling, is examined briskly, and it is replacing for the material of an inorganic system.

[0004] Although sensitivity, improvement in endurance, etc. are mentioned as a technical problem in the electrophotography photo conductor of an organic system, using a condensation imidazole pigment as carrier generating matter contained in a photosensitive layer as a policy to these is proposed.

[0005] For example, the technology of using a screw benzimidazole sulfur-ized perylene compound and a perylene compound unsymmetrical to U.S. JP,4714666,B at a screw naphth imidazole perylene compound and JP,57-192958,A at a halogenation screw benzimidazole perylene compound and JP,3-42670,A is opened to a screw benzimidazole perylene compound and U.S. JP,4792508,B at U.S. JP,3972717,B and JP,59-59686,A.

[0006] In manufacture of these condensation imidazole pigments, the dehydration condensation reaction which used the corresponding aromatic carboxylic-acid anhydride and the aromatic diamino compound as the raw material has been performed from the former. Generally in such a manufacturing method, a quinoline, alpha-crawl naphthalene, O-dichlorobenzene, N-methyl pyrrolidone, N,N-dimethylformamide, ethylene glycol, a tetralin, a nitrobenzene, 1 and 2, 4-trichlorobenzene, a glacial acetic acid, water, etc. are used as a reaction solvent.

[0007] However, since a reaction advances under a remarkable high temperature service, generally it is necessary to heat this reaction at 200 degrees C or more. Moreover, depending on a reaction solvent, a dehydration condensation reaction hardly advances, but, in such a case, a dehydration catalyst is used. As a dehydration catalyst, a zinc chloride, zinc acetate, a zinc oxide, an acetic acid, a hydrochloric acid, p-toluenesulfonic acid, etc. are effective. However, when a dehydration catalyst is used, it is required to heat at 190-200 degrees C or more.

[0008] For example, a 205 degrees C [which used N-methyl pyrrolidone as the reaction solvent at JP,59-59686,A] reaction, A 250 degrees C [which used alpha-crawl naphthalene as the reaction solvent at U.S. JP,4792508,B] reaction, A 210 degrees C which used the acetic acid as the reaction solvent at JP,61-275848,A] reaction, The example of a 200-300 degrees C which used N,N-dimethylformamide as the reaction solvent at JP,63-291061,A reaction, and a 195-202 degrees C [which used N-methyl pyrrolidone as the reaction solvent at JP,3-42670,A, and made the zinc acetate dihydrate the dehydration catalyst] reaction is reported. *US 172126*

[0009] Heating of 200 degrees C or more is required for each of these, and the reaction did not advance as a matter of fact at low temperature from this.

[0010]

[Problem(s) to be Solved by the Invention] Thus, since the reaction in an elevated temperature was required for manufacture of a condensation imidazole pigment, it was easy to mix the impurity produced by side reaction or the decomposition reaction, and the fall of the electrophotography sensitivity of a resultant was not avoided as a result.

[0011] Moreover, in order to perform pyrogenetic reaction 200 degrees C or more, an expensive heating facility of circulation of heating oil etc. is needed required [the reaction iron pot which consists of the heat-resistant quality of the material]. In addition, as for alpha-crawl naphthalene, a quinoline, etc. which are a reactant high solvent, the plant-and-equipment investment on a work environment is needed from an odor and the field of detrimental nature.

[0012] this invention is made based on the above situations, and the purpose is in offering the new manufacture method by the low-temperature reaction of a condensation imidazole pigment.

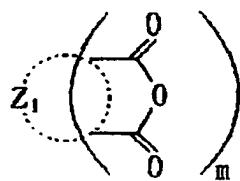
[0013] Moreover, the purpose of this invention manufactures a highly efficient condensation imidazole pigment, and offers a high sensitivity electrophotography photo conductor.

[0014]

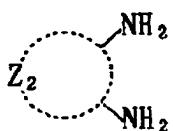
[Means for Solving the Problem] The following composition can attain the above-mentioned purpose of this invention.

[0015] (1) The manufacture method of the organic pigment characterized by using the compound expressed with a general

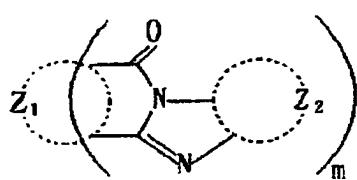
[1]



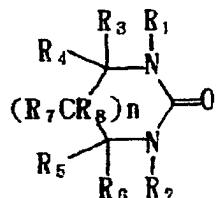
[2]



[3]



[4]



As for the inside Z1 and Z2 of a formula, the aromatic ring which is not replaced [substitution and] is expressed and m expresses 1 or 2. Moreover, R1-R8 express a hydrogen atom, an alkyl, and an aryl group, and n expresses 0 or 1.

[Translation done.]

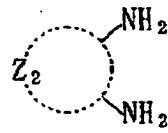
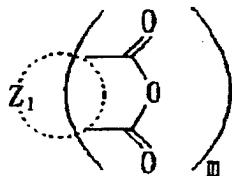
formula [4] as a reaction solvent by the dehydration condensation reaction of an aromatic diamino compound expressed with the aromatic carboxylic-acid anhydride expressed with a general formula [1], and a general formula [2] in case the condensation imidazole pigment of a general formula [3] is manufactured.

[0016]

[Formula 3]

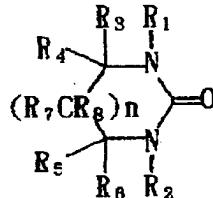
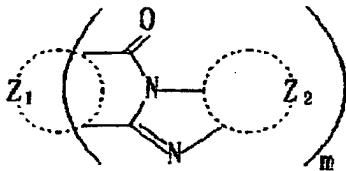
[1]

[2]



[3]

[4]



[0017] As for the inside Z1 and Z2 of a formula, the aromatic ring which is not replaced [substitution and] is expressed and m expresses 1 or 2. Moreover, R1-R8 express a hydrogen atom, an alkyl, and an aryl group, and n expresses 0 or 1.

[0018] (2) Z1 in a general formula [1] is a perylene ring or (N-alkyl) a perylene. - 3, 4, the manufacture method of the organic pigment the above-mentioned publication which is - dicarboxyimide ring.

[0019] (3) The electrophotography photo conductor which comes to contain the organic pigment characterized by using the compound expressed with a general formula [4] as a reaction solvent by the dehydration condensation reaction of an aromatic diamino compound expressed with the aromatic carboxylic-acid anhydride expressed with a general formula [1], and a general formula [2] in case the condensation imidazole pigment of a general formula [3] is manufactured.

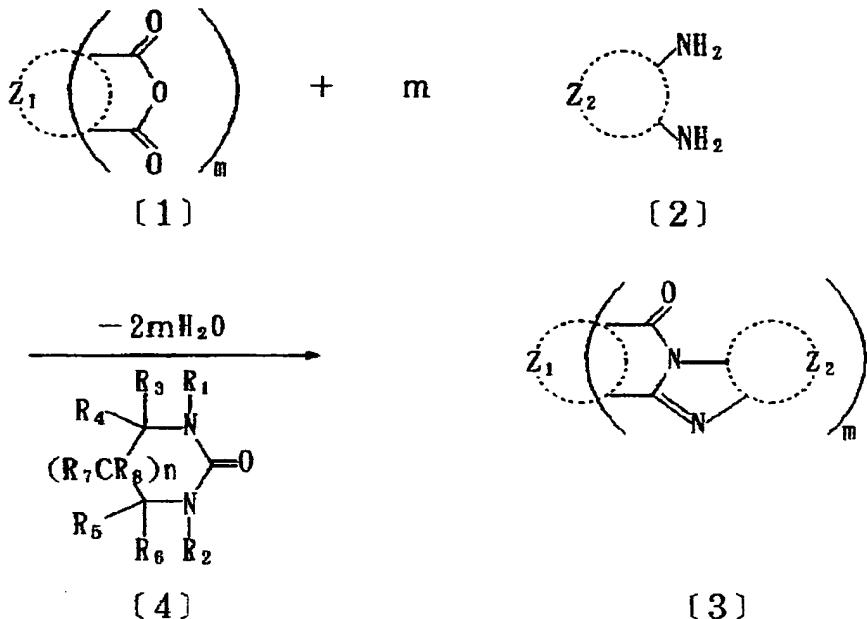
[0020] It is desirable to use zinc acetate or a zinc chloride especially as a dehydration catalyst, and reaction temperature can be made into 150 degrees C or less. For this reason, it is not necessary to make a reaction iron pot into the heat-resistant quality of the material and, and since it is temperature sufficient by steam heating, it is not necessary to introduce an expensive heating facility. Moreover, generally, since there are few odors, the compound expressed with a general formula [4] becomes good [a work environment]. Thus, the sensitivity performance of the electrophotography photo conductor produced using the pigment with which an installation cost and its operating cost were remarkably mitigable in the reaction in low temperature being possible in the manufacturing method of this invention with the bird clapper, and also side reaction and the decomposition reaction stopped being able to happen easily, and were obtained improves.

[0021] A reaction formula (1) can express the dehydration condensation reaction of this invention.

[0022]

[Formula 4]

反応式 (1)



[0023] Z₁ and Z₂ express among the formula the aromatic ring which is not replaced [substitution and], and the desirable things of Z₁ are the benzene ring, a naphthalene ring, a pyrene ring, a perylene ring, a coronene ring, sulfur-ized perylene rings, and these dicarboxyimide derivatives. Especially desirable things are a pyrene ring, a perylene ring, a pyrene (N-alkyl)-2, 3-dicarboxyimide ring, a perylene (N-alkyl)-3, and 4-dicarboxyimide ring especially.

[0024] The desirable things of Z₂ are the benzene ring and a naphthalene ring. As these substituents, an alkyl, an aryl, an alkoxy ** aralkyl, nitroglycerine, a cyano group, a halogen, etc. can be mentioned, and they are an alkyl, an aryl, and an aralkyl preferably.

[0025] m expresses 1 or 2.

[0026] Moreover, among a formula, R₁-R₈ express the hydrogen atom, the alkyl, and the aryl group, and they are a hydrogen atom and a four or less-carbon number alkyl group preferably. n expresses 0 or 1. As a desirable compound, R₁ and R₂ can mention especially the 1 and 3-dimethyl-2-imidazolidinone whose n a methyl group, and R₃-R₆ are hydrogen atoms, and is 0.

[0027] Although the amount of the aromatic diamino compound to an aromatic carboxylic-acid anhydride may be arbitrary according to the purpose in this reaction, it becomes the range of 0.1 to 100Eq preferably, and the range of 1 to 10Eq is especially desirable.

[0028] As a dehydration catalyst, although a zinc chloride, zinc acetate, a zinc oxide, an acetic acid, a hydrochloric acid, p-toluenesulfonic acid, etc. can be used, especially, zinc acetate and a zinc chloride are desirable and especially zinc acetate is desirable. The charge of the dehydration catalyst to an acid anhydride has desirable 0.1 to 100Eq, and its 1 to 6Eq is especially desirable.

[0029] Moreover, the range of reaction temperature of 80-180 degrees C is desirable.

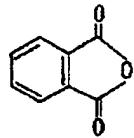
[0030]

[Function] The typical example of an aromatic carboxylic-acid anhydride expressed with a useful general formula [1] in this invention is raised.

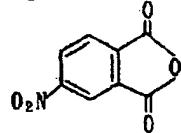
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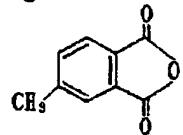
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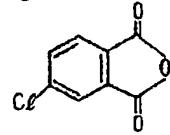
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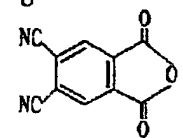
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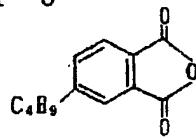
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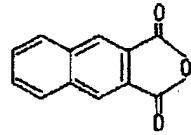
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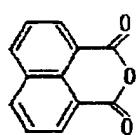
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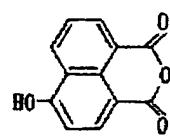
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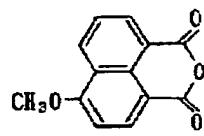
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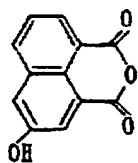


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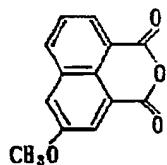


[0032]
[Formula 6]

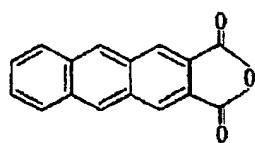
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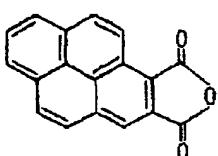
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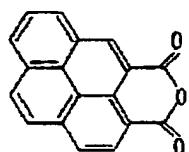
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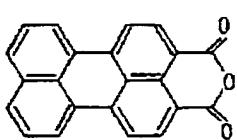
1 - 14



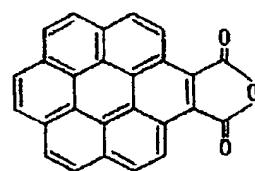
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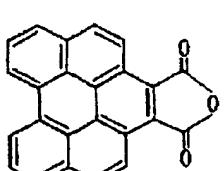
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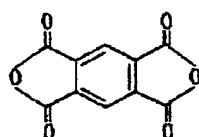
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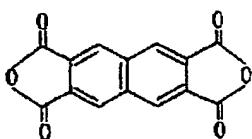
1 - 18



1 - 19

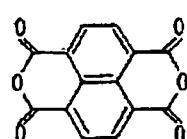


1 - 20

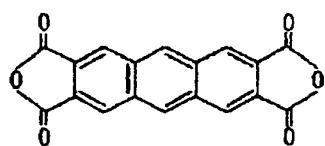


[0033]
[Formula 7]

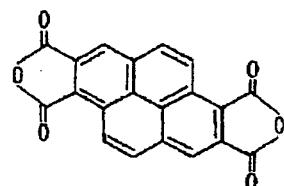
1 - 21



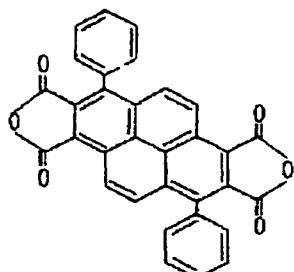
1 - 22



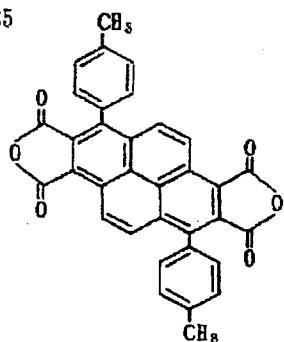
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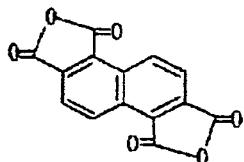
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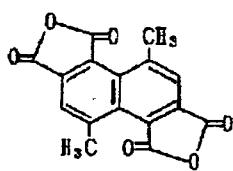
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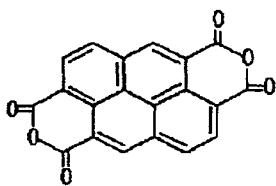
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1 - 27

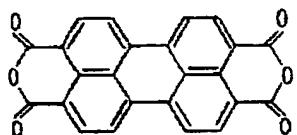


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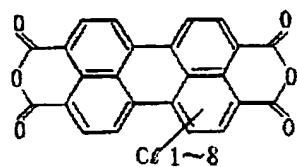


[0034]
[Formula 8]

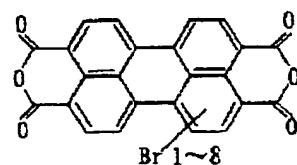
1 - 29



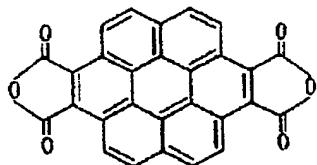
1 - 30



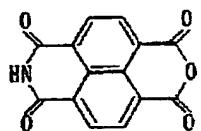
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1 - 32

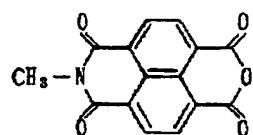


1 - 33

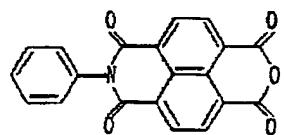


[0035]
[Formula 9]

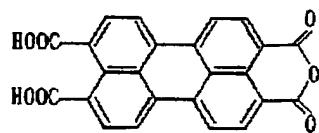
1 - 34



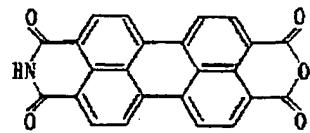
1 - 35



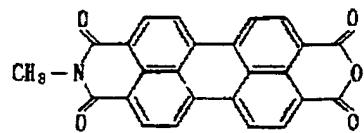
1 - 36



1 - 37

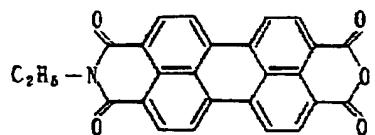


1 - 38

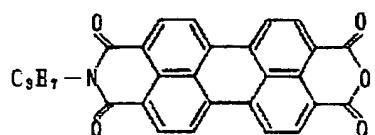


[0036]
[Formula 10]

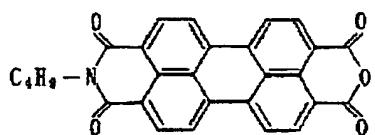
1 - 39



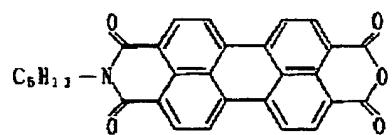
1 - 40



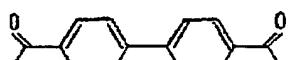
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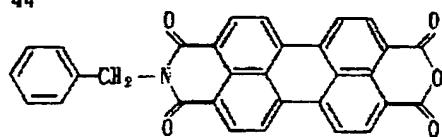
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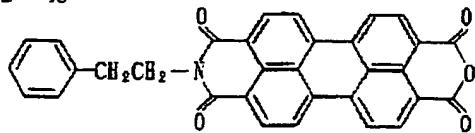
1 - 43



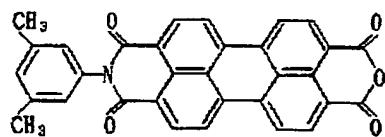
[0037]
[Formula 11]
1 - 44



1 - 45

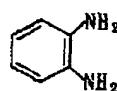


1 - 46

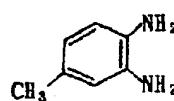


[0038] The typical example of an aromatic diamine compound expressed with a useful general formula [2] in this invention is raised.

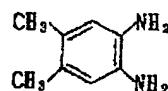
[0039]
[Formula 12]
2 - 1



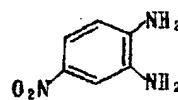
2 - 2



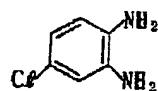
2 - 3



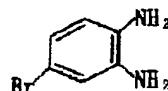
2 - 4



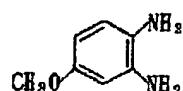
2 - 5



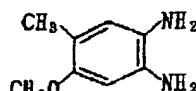
2 - 6



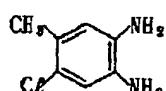
2 - 7



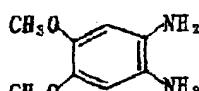
2 - 8



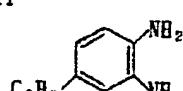
2 - 9



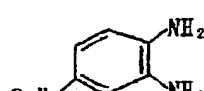
2 - 10



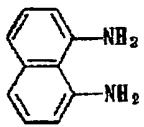
2 - 11



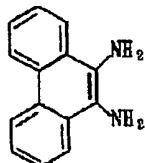
2 - 12



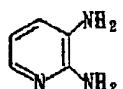
[0040]
[Formula 13]
2 - 15



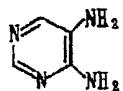
2 - 16



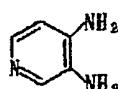
2 - 17



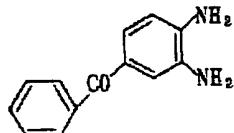
2 - 18



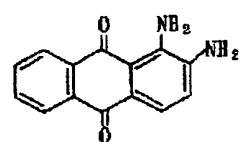
2 - 19



2 - 20



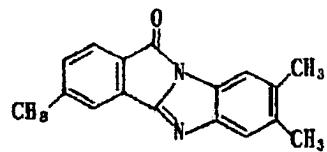
2 - 21



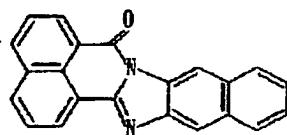
[0041] Next, the condensation imidazole pigment of a general formula [3] shows some of the examples of representation, although dehydration condensation is carried out in the arbitrary combination of the compound of a general formula [1] and a general formula [2].

[0042]
[Formula 14]

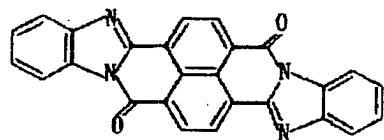
3 - 1



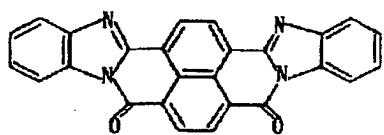
3 - 2



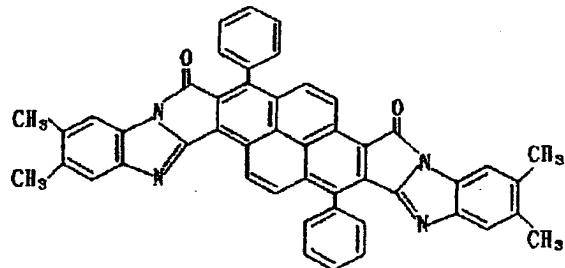
3 - 3



3 - 4

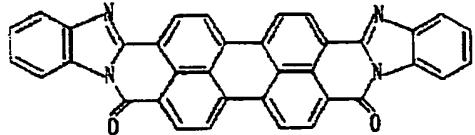


3 - 5

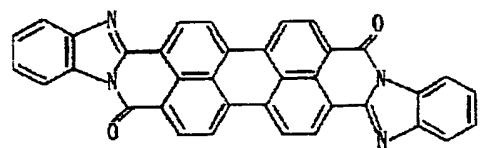


[0043]
[Formula 15]

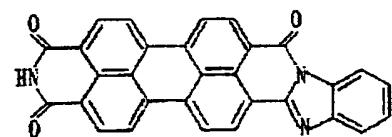
3 - 6



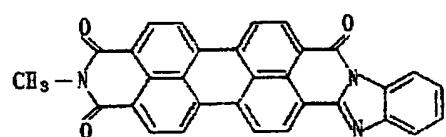
3 - 7



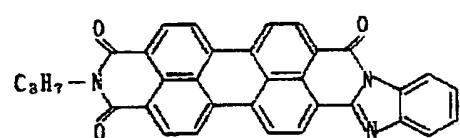
3 - 8



3 - 9

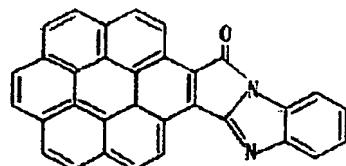


3 - 10

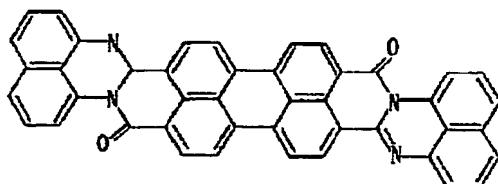


[0044]
[Formula 16]

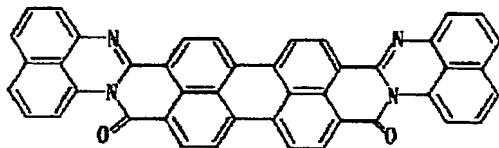
3 - 11



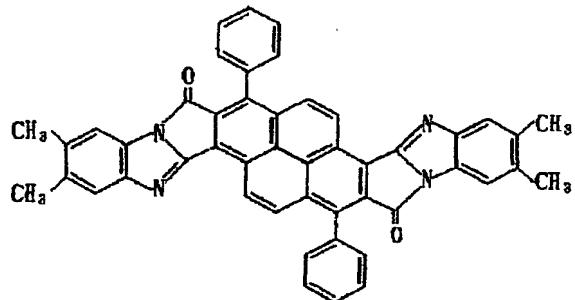
3 - 12



3 - 13



3 - 14

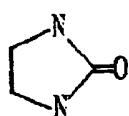


[0045] Next, the example of a compound expressed with a general formula [4] is shown.

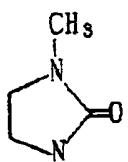
[0046]

[Formula 17]

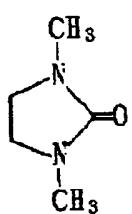
4 - 1



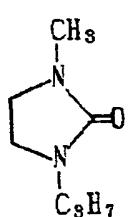
4 - 2



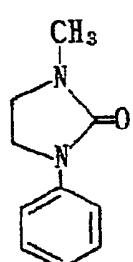
4 - 3



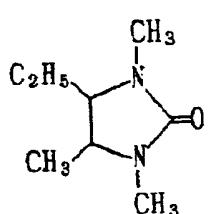
4 - 4



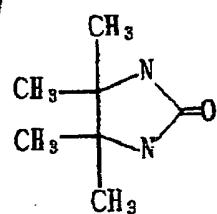
4 - 5



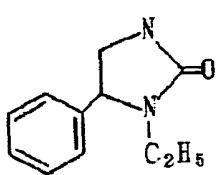
4 - 6



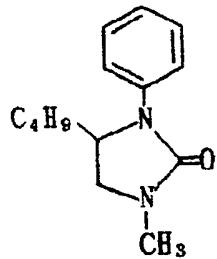
4 - 7



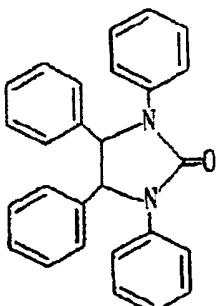
4 - 8



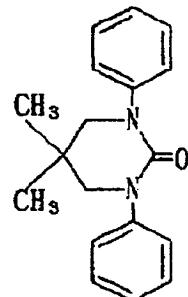
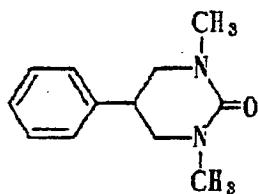
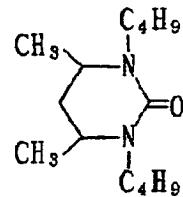
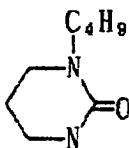
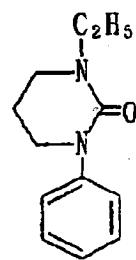
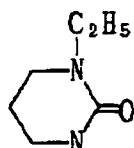
4 - 9



4 - 10



[0047]
[Formula 18]



[0048] The advance situation of a synthetic reaction can be checked by measuring the visible absorption spectrum of the system of reaction. Since the raw material compound and product pigment in a reaction of this invention generally dissolve in a concentrated sulfuric acid, it is useful to melt the sample sampled from the system of reaction to a sulfuric acid, and to measure an absorption spectrum. The absorption maximum of a raw material compound is in a short wavelength side rather than the absorption maximum of a product, and the advance condition of a reaction is checked by carrying out the comparison trace of both maximum peak.

[0049] When using the organic pigment obtained by this invention as carrier generating matter of an electrophotography photo conductor, the gestalt various in the composition of a photo conductor is known. Although the pigment of this invention can be used in any [the] case, it is desirable to consider as the functional discrete-type photo conductor of a laminating type or distributed type. In this case, it becomes lamination usually like drawing (a) - (f). The lamination shown in drawing (a) forms the carrier generating layer 2 on the conductive base material 1, the laminating of the carrier transporting bed 3 is carried out to this, a photosensitive layer 4 is formed, and this drawing (b) forms photosensitive-layer 4' which made reverse these carrier generating layers 2 and carrier transporting beds 3. This drawing (c) forms an interlayer 5 between the photosensitive layer 4 of the lamination of (a), and the conductive base material 1, and this drawing (d) forms an interlayer 5' between photosensitive-layer 4' of the lamination of (b), and the conductive base material 1. The lamination of this drawing (e) forms photosensitive-layer 4" containing the carrier generating matter 6 and the carrier transportation matter 7, and this drawing (f) forms an interlayer 5 between such photosensitive-layer 4" and the conductive base material 1. Drawing (a) In the composition of - (f), a protective layer can be further prepared in the maximum surface.

[0050] In formation of a photosensitive layer, the method of applying the solution in which it is independent or the carrier generating matter or the carrier transportation matter was dissolved with the binder or the additive is effective.

[0051] However, generally for a low reason, the solubility of the pigment of this invention becomes effective [the method of applying the liquid which carried out particle distribution of the pigment into the suitable dispersion medium using distributed equipments, such as an ultrasonic disperser, a ball mill, a sand mill, and a homomixer, in such a case, or the method of carrying out vacuum evaporation under reduced pressure and making a carrier generating layer form].

[0052] Large arbitrary things can be used as the solvent used for formation of a photosensitive layer, or a dispersion medium.

[0053] For example, a methyl ethyl ketone, methyl isopropyl ketone, a cyclohexanone, a tetrahydrofuran, an acetone, a methyl isobutyl ketone, dichloromethane, dichloroethane, trichloroethane, chloroform, a methanol, ethanol, propanol, a butanol, a butylamine, ethylenediamine, N,N-dimethylformamide, ethyl acetate, butyl acetate, a methyl Cellosolve, an ethyl Cellosolve, an ethylene glycol wood ether, toluene, a xylene, an acetophenone, etc. are mentioned.

[0054] Moreover, in formation of a photosensitive layer, although things arbitrary as a binder can be chosen when using a binder, it is especially hydrophobic and the macromolecule polymer which has film organization potency is desirable. Although the following can be mentioned as such a polymer, it is not limited to these.

[0055] Polycarbonate polycarbonate Z resin acrylic resin methacrylic resin polyvinyl chloride polyvinylidene chloride polystyrene styrene-butadiene-polymer polyvinyl acetate poly BINIRUHO A RUMARUPORIBINIRUBUCHIRARU polyvinyl-butyl polyvinyl-carbazole styrene-alkyd-resin silicone resin silicone-alkyd-resin silicone-alkyd resin The rate of a polyester phenol resin polyurethane epoxy resin vinylidene-chloride-acrylonitrile-copolymer vinyl-chloride-vinyl-acetate-copolymer vinyl chloride-vinyl acetate-maleic-anhydride copolymer binder and the carrier generating matter receives the binder 100 weight section. The carrier generating matter 10 - the 1000 weight sections are desirable, and especially the further 50 to 400 weight section is desirable. Moreover, as for the rate of a binder and the carrier transportation matter, it is desirable to consider as the carrier transportation matter 10 - the 500 weight sections to the binder 100 weight section.

[0056] Arbitrary things can be chosen as matter used for an interlayer and a protective layer. Although the following example can be given besides the binder mentioned to the above-mentioned carrier generating layer and carrier transporting beds, it is not limited to these.

[0057] Polyamide resin Nylon ethylene-vinyl acetate copolymer ethylene-vinyl acetate-maleic-anhydride copolymer ethylene - as vinyl acetate-methacrylic-acid copolymer polyvinyl alcohol cellulosic melamine resin epoxy resin silicone resin carrier transportation matter Although the thing of ***** can be used As a typical thing, for example An oxazole, an OKISA diazole, a thiazole, The compound which has the nitrogen-containing heterocycle nucleus represented by thiadiazole, the imidazole, etc. and its condensed-ring nucleus, The compound of the poly aryl alkane system, a pyrazoline system compound, a hydrazone system compound, A triaryl amine compound, a styryl system compound, a styryl triphenylamine system compound, beta-phenyl styryl triphenylamine system compound, a butadiene system compound, a hexa trien system compound, a carbazole system compound, a condensation polycyclic compound, etc. are mentioned.

[0058] Although carrier generating layer thickness is set to 0.01-20 micrometers, further 0.05-5 micrometers is desirable.

Although thickness of a carrier transporting bed is set to 1-100 micrometers, further 5-50 micrometers is desirable.

[0059] As a conductive base material, a metal plate and a metal drum are used, and also the thing which comes to prepare the thin layer of metals, such as conductive compounds, such as conductive polymer and indium oxide, or aluminum, and palladium, by technique, such as an application, vacuum evaporation, and a lamination, on bases, such as paper and plastics, can be used.

[0060] The photo conductor using the pigment of this invention can be considered as the above composition.

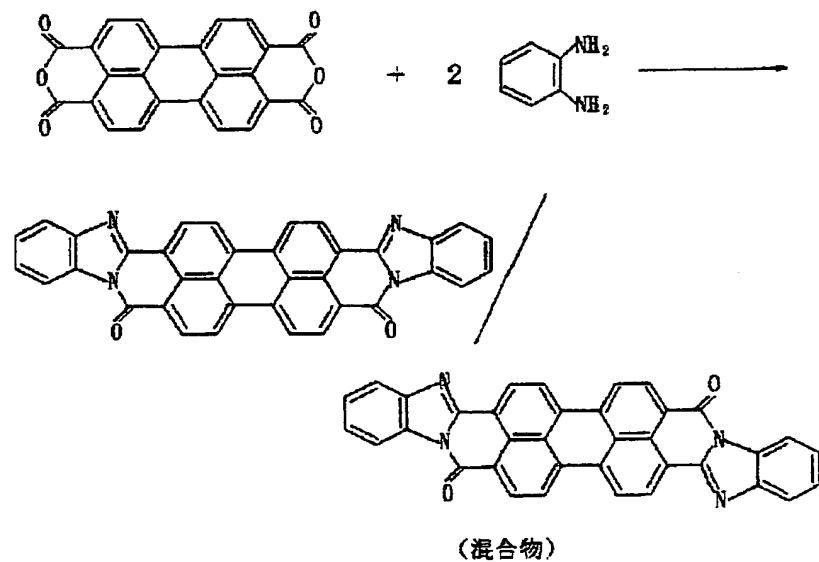
[0061]

[Example] Although an example is given and this invention is explained in detail hereafter, the mode of this invention is not limited to this.

[0062] Example 1 reaction formula (2) is followed and it is a perylene. - 1 and 3-dimethyl-2-imidazolidinone 118ml was mixed as 5.60g (anhydrous) of zinc acetate, and a reaction solvent as 3, 4, 9, 6.0g [of 10-tetrapod carboxylic-acid 2 anhydrides], and O-phenylenediamine 6.61g, and a dehydration catalyst, and it was made to react at 150 degrees C for 7 hours. After it separated the crystal which deposited after radiation cooling and dilute hydrochloric acid washed, N,N-dimethylformamide and water washed. Stoving was carried out and 7.92g (97% of yield) of crystals was obtained. Let this be a sample 1.

[0063]

[Formula 19]
反応式 (2)



[0064] When example 2 dehydration catalyst was changed into the zinc chloride (anhydrous) from zinc acetate (anhydrous) and also having been reacted like the example 1, 10 hours was required till the reaction end. Washing operation was performed like the example 1 and 7.76g (95% of yield) was obtained. Let this be a sample 2.

[0065] The example of comparison 1 reaction solvent was changed into N,N-dimethylformamide, and also it reacted at 150 degrees C like the example 1. However, advance of a reaction was slow and even the reaction of 20 hours did not reach the absorption spectrum of the specified substance completely in evaluation by the visible absorption spectrum.

[0066] Made the example of comparison 2 reaction solvent into N,N-dimethylformamide, and the dehydration catalyst was used as the zinc chloride (anhydrous), and also it reacted at 150 degrees C like the example 1. As well as the example 2 of comparison, the reaction was slow and, as for the specified substance, even the reaction of 20 hours was not obtained.

[0067] The example of comparison 3 reaction solvent was changed into N-methyl pyrrolidone, and also it reacted at 150 degrees C like the example 1. Even the reactions of 20 hours of generation of the specified substance were few.

[0068] The example of comparison 4 reaction solvent was changed into alpha-crawl naphthalene, and also it reacted at 150 degrees C like the example 1. The specified substance hardly generated even the reaction of 20 hours.

[0069] Changed the example of comparison 5 reaction solvent into alpha-crawl naphthalene, and a dehydration catalyst was not used, and also it reacted at 150 degrees C like the example 1. At all, although the reaction did not progress but reaction temperature was made into 200 degrees C, the reaction was very slow and was not ended in 20 hours. Then, when reaction temperature was made into 250 degrees C, the reaction was completed in 4 hours. It cooled radiationally, precipitated crystal was separated, alpha-crawl naphthalene and the methanol washed, and 7.68g (94% of yield) of specified substance was obtained. Let this be a comparison sample (1).

[0070] The example of comparison 6 reaction solvent was changed into the quinoline, and also it reacted at 150 degrees C like the example 1. However, a reaction hardly advanced. Then, when it was made to react at 230 degrees C, it ended in 4.5 hours. It cooled radiationally, a quinoline, chloroform, and the methanol repeated and washed the crystal which deposited, and 7.5g (92% of yield) of specified substance was obtained. Let this be a comparison sample (2).

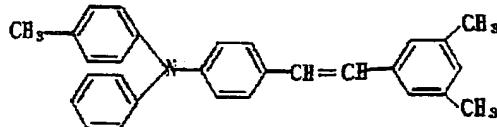
[0071] Only when 1 of this invention and 3-dimethyl-2-imidazolidinone is used for a reaction solvent as mentioned above, reaction temperature can be made into 150 degrees C, and unless it is at least 200 degrees C or more in the reaction solvent known conventionally, a reaction does not advance promptly.

[0072] According to the following application, the electrophotography photo conductor was produced and evaluated using the sample 1 of the obtained this invention, the sample 2, the comparison sample for comparison (1), and the comparison sample (2).

[0073] 1 weight section was used for the sample 1 of this invention obtained in the application 1 example 1, the 0.2 weight section and the methyl-ethyl-ketone 30 weight section were used for the polyvinyl butyral "S REKKU BL-S" (Sekisui Chemical Co., Ltd. make), the distributed liquid distributed and obtained by the sand mill was applied with the wire bar on the polyester base which deposited aluminum, and the 0.4-micrometer carrier generating layer was formed. The liquid which dissolved the carrier transportation matter 1 section of the following structure expression (T-1) and the polycarbonate resin "Z-200" (Mitsubishi **** chemistry company make) 1.3 section in the dichloroethane 8 section on it was applied using the blade application machine, the 30-micrometer carrier transporting bed was formed, and the photo conductor was obtained. Let this be a photo conductor 1.

[0074]

[Formula 20]
(T-1)



[0075] Application 2 sample 1 was replaced with the sample 2 obtained in the example 2, and also the photo conductor was obtained like the application 1. Let this be a photo conductor 2.

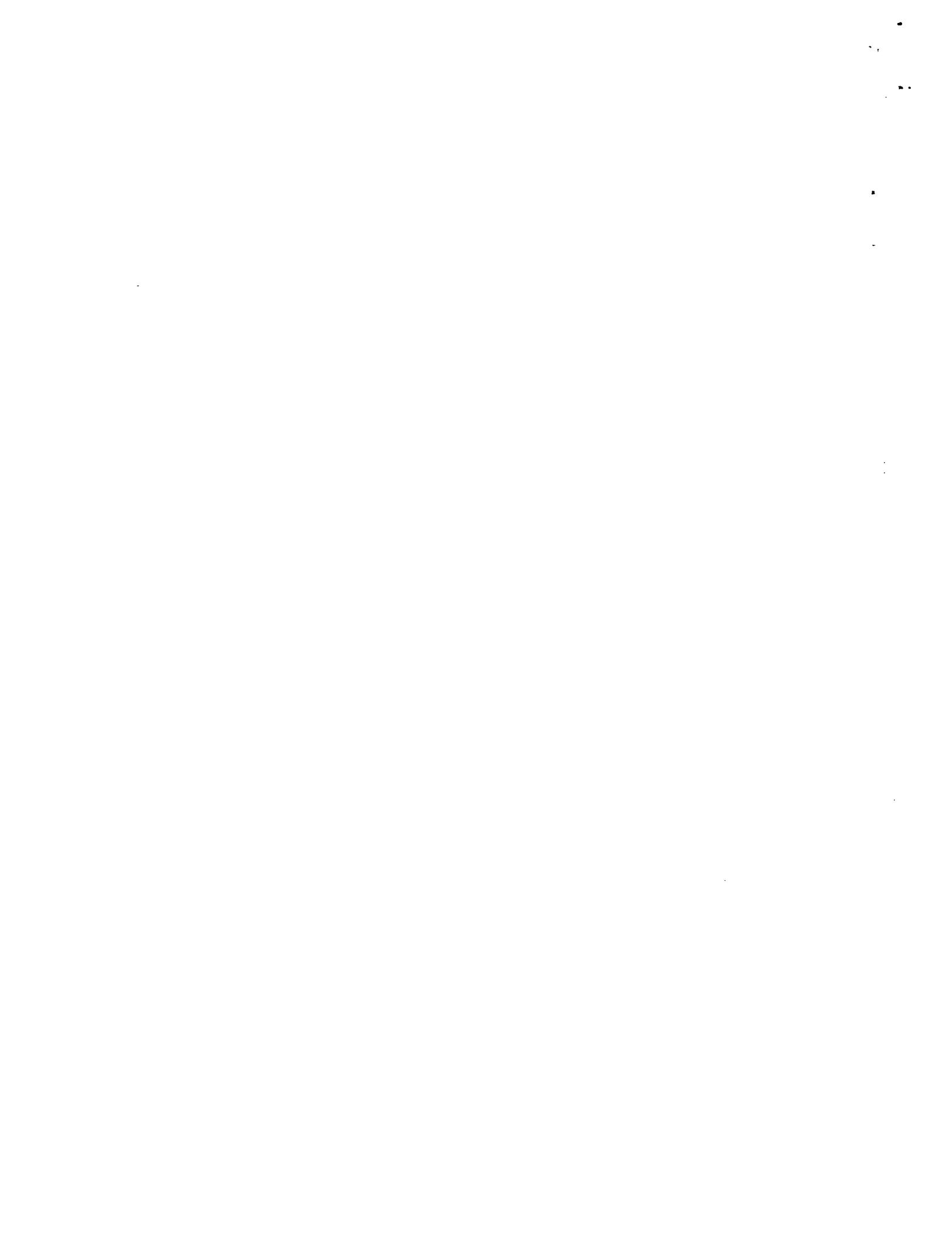
[0076] One to comparison application 2 sample 1 was replaced with the comparison sample (1) and the comparison sample (2), and also the comparative photo conductor was obtained like the application 1. Let this be a comparison photo conductor (1) and a comparison photo conductor (2), respectively.

[0077] These photo conductors performed the following evaluations using paper analyzer EPA-8100 (the Kawaguchi electrical machinery company make). First, corona electrical charging for 5 seconds was performed on -6kV conditions, it asked for the surface potential V_a immediately after electrification, and the surface potential V_i after the neglect during 5 seconds, and exposure from which a surface illuminance is continuously set to 2 (lux) was performed, and light exposure $E_{600/100}$ were calculated. required to reduce surface potential from -600V to -100V] Moreover, it asked for the rate D of a dark decay from the formula of $D = 100 (V_a - V_i) / V_a (\%)$.

[0078] The result was shown in Table 1.

[0079]
[Table 1]

サンプル	V_a (V)	V_i (V)	D (%)	$E_{600/100}$ (lux·sec)
感光体1	-1540	-1260	18.2	3.5
感光体2	-1500	-1240	17.3	3.7
比較感光体(1)	-1310	-1030	21.4	5.5
比較感光体(2)	-1340	-1020	23.9	5.1



[0080] Three to example 10 reaction formula (1) is followed, and it is 1 and 3-dimethyl-2-imidazolidinone in 3Eq (anhydrous) of zinc acetate as 2Eq of an aromatic diamino compound [2], and a dehydration catalyst to 0.01 mols of an aromatic carboxylic acid anhydride [1]. It was made to react in 100ml in 150 degrees C and 8 hours. After it separated the crystal which deposited after radiation cooling and dilute hydrochloric acid washed, N,N-dimethylformamide and water washed and the condensation imidazole pigment [3] was obtained after stoving.

[0081] The result was summarized in Table 2.

[0082]

[Table 2]

	原料(1)	原料(2)	生成物(3)	収率(%)
実施例 3	例示化合物 1 - 8	例示化合物 2 - 13	例示化合物 3 - 2	85.0
実施例 4	例示化合物 1 - 17	例示化合物 2 - 1	例示化合物 3 - 11	82.3
実施例 5	例示化合物 1 - 21	例示化合物 2 - 1	例示化合物 3 - 3 例示化合物 3 - 4	89.5
実施例 6	例示化合物 1 - 24	例示化合物 2 - 3	例示化合物 3 - 5 例示化合物 3 - 14	89.0
実施例 7	例示化合物 1 - 37	例示化合物 2 - 1	例示化合物 3 - 8	93.3
実施例 8	例示化合物 1 - 38	例示化合物 2 - 1	例示化合物 3 - 9	91.6
実施例 9	例示化合物 1 - 40	例示化合物 2 - 1	例示化合物 3 - 10	95.1
実施例 10	例示化合物 1 - 29	例示化合物 2 - 15	例示化合物 3 - 12 例示化合物 3 - 13	90.0

[0083] The reaction for which the temperature of 200 degrees C or more was required can make it go on with sufficient yield at the low temperature of 150 degrees C or less conventionally in the manufacturing method of this invention as mentioned above. Moreover, the condensation imidazole pigment manufactured on such quiet conditions shows the sensitivity performance which was excellent as carrier generating matter of an electrophotography photo conductor.

[0084]

[Effect of the Invention] Conventionally, since the reaction in an elevated temperature was required for manufacture of a condensation imidazole pigment, it was easy to mix the impurity produced by side reaction or the decomposition reaction, and the fall of the electrophotography sensitivity of a resultant was not avoided as a result.

[0085] Moreover, in order to perform pyrogenetic reaction 200 degrees C or more, an expensive heating facility of circulation of heating oil etc. is needed required [the reaction iron pot which consists of the heat-resistant quality of the material]. In addition, as for alpha-crawl naphthalene, a quinoline, etc. which are a reactant high solvent, the plant-and-equipment investment on a work environment is needed from an odor and the field of detrimental nature.

[0086] By this invention, the new manufacture method by the low-temperature reaction of a condensation imidazole pigment can be offered.

[0087] Moreover, a highly efficient condensation imidazole pigment can be manufactured and a high sensitivity electrophotography photo conductor can be offered.

[Translation done.]

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